

checked by IT  
11/16/15

## MEMORANDUM

**TO:** Mr. Addison Rice  
Anderson, Mulholland and Associates

**DATE:** November 11, 2015

**FROM:** R. Infante

**FILE:** 1510351C

**RE:** Data Validation  
Air samples  
SDG: 1510351C

### SUMMARY

Full validation was performed on the data for several gas samples analyzed for methane by ASTM method D-1946-modified. The samples were collected at the Bristol Myer Squib-Building 6 VI facility, Humacao, PR site on October 18, 2015 and submitted to Eurofins Air Toxics, Inc. of Folsom, California that analyzed and reported the results under delivery groups (SDG) 1510351C.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006; and the QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

### SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B30IA-1 101715	1510351C-01A	10/18/2015	Air	Methane
B30IA-2 101715	1510351C-02A	10/18/2015	Air	Methane
B30IA-3 101715	1510351C-03A	10/18/2015	Air	Methane
B30IA-4 101715	1510351C-04A	10/18/2015	Air	Methane
B30IA-4D 101715	1510351C-05A	10/18/2015	Air	Methane
B30IA-5 101715	1510351C-06A	10/18/2015	Air	Methane
B42IA-1 101715	1510351C-07A	10/18/2015	Air	Methane
B42IA-2 101715	1510351C-08A	10/18/2015	Air	Methane
B42IA-3 101715	1510351C-09A	10/18/2015	Air	Methane
B3042AA 101715	1510351C-10A	10/18/2015	Air	Methane
B8IA-2 101715	1510351C-11A	10/18/2015	Air	Methane
B8IA-2D 101715	1510351C-12A	10/18/2015	Air	Methane
B8AA-1 101715	1510351C-13A	10/18/2015	Air	Methane

## REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Canister cleaning certification criteria
- Surrogate spike recovery
- Internal standard performance and retention times
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

## DISCUSSION

### Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form except for the following:

- Sample B30IA-1 101715F was not analyzed.

### Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

### Initial and Continuing Calibrations

#### Methane by ASTM method D-1946 (modified)

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

### Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

Summa canister met cleaning certification criteria.

No trip/field blank analyzed with this data package.

### Laboratory/Field Duplicate Results

Field/laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of + 25 % for analytes 5 x SQL.

### LCS/LCSD Results

#### Methane

LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

### Quantitation Limits and Sample Results

Dilutions were not performed (see worksheet).

Calculations were spot checked.

### Certification

The following samples 1510351C-01A; 1510351C-02A; 1510351C-03A; 1510351C-04A; 1510351C-05A; 1510351C-06A; 1510351C-07A; 1510351C-08A; 1510351C-09A; 1510351C-10A; 1510351C-11A; 1510351C-12A; and 1502113B-13A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

  
Rafael Infante  
Chemist License 1888





Air Toxics

Client Sample ID: B30IA-1 101715

Lab ID#: 1510351C-01A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9102205	Date of Collection: 10/18/15 11:00:00 A
Dil. Factor:	1.66	Date of Analysis: 10/22/15 02:18 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00023

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B301A-2 101715

Lab ID#: 1510351C-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9102206	Date of Collection: 10/18/15 11:26:00 A
Dil. Factor:	1.67	Date of Analysis: 10/22/15 03:16 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00032

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30IA-3 101715

Lab ID#: 1510351C-03A

**NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946**

File Name: 9102207  
Dil. Factor: 1.72

Date of Collection: 10/18/15 11:59:00 A  
Date of Analysis: 10/22/15 03:37 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00029

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30IA-4 101715

Lab ID#: 1510351C-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102208  
Dil. Factor: 1.60

Date of Collection: 10/18/15 11:38:00 A  
Date of Analysis: 10/22/15 04:16 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00016	0.00028

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B30IA-4D 101715

Lab ID#: 1510351C-05A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102209  
Dil. Factor: 1.51

Date of Collection: 10/18/15 11:38:00 A  
Date of Analysis: 10/22/15 04:45 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00015	0.00028

Container Type: 6 Liter Summa Canister (100% Certified)







Air Toxics

Client Sample ID: B30IA-5 101715

Lab ID#: 1510351C-06A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102210  
Dil. Factor: 1.69

Date of Collection: 10/18/15 11:32:00 A  
Date of Analysis: 10/22/15 05:08 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00027

Container Type: 6 Liter Summa Canister (100% Certified)





## Air Toxics

Client Sample ID: B42IA-1 101715

Lab ID#: 1510351C-07A

### NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102211  
Dil. Factor: 1.68

Date of Collection: 10/18/15 12:38:00 P  
Date of Analysis: 10/22/15 06:15 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00017	0.00020

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B42IA-2 101715

Lab ID#: 1510351C-08A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102212  
Dil. Factor: 1.57

Date of Collection: 10/18/15 7:54:00 AM  
Date of Analysis: 10/22/15 06:39 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00016	0.00020

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B42IA-3 101715

Lab ID#: 1510351C-09A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102213  
Dil. Factor: 1.46

Date of Collection: 10/18/15 7:52:00 AM  
Date of Analysis: 10/22/15 07:19 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00015	0.00020

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B3042AA

Lab ID#: 1510351C-10A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9102214	Date of Collection: 10/18/15 1:45:00 PM
Dil. Factor:	1.88	Date of Analysis: 10/22/15 07:49 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00019	0.00020

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B8IA-2 101715

Lab ID#: 1510351C-11A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102215  
Dil. Factor: 1.57

Date of Collection: 10/18/15 11:45:00 A  
Date of Analysis: 10/22/15 09:04 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00016	0.00021

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B8IA-2D 101715

Lab ID#: 1510351C-12A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102216  
Dil. Factor: 1.77

Date of Collection: 10/18/15 11:45:00 A  
Date of Analysis: 10/22/15 09:25 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00018	0.00022

Container Type: 6 Liter Summa Canister (100% Certified)





Air Toxics

Client Sample ID: B8AA-1 101715

Lab ID#: 1510351C-13A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: 9102217  
Dil. Factor: 1.58

Date of Collection: 10/18/15 11:45:00 A  
Date of Analysis: 10/22/15 09:51 PM

Compound	Rpt. Limit (%)	Amount (%)
Methane	0.00016	0.00020

Container Type: 6 Liter Summa Canister (100% Certified)







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FOLSOM, CA 95630-4719  
(916) 985-1000 FAX (916) 985-1020

Page 1 of 2

Project Manager Terry Taylor

Collected by: (Print and Sign) David Lindstrand

Company AMA1 Email \_\_\_\_\_

Address 2700 Westchester City Purchase State NY zip 10577

Phone 914-251-0400 Fax \_\_\_\_\_

Project Info:

P.O. # \_\_\_\_\_

Project # \_\_\_\_\_

Project Name BMS V1

Turn Around Time:

☐ Normal

☒ Rush

3-Day

Lab Use Only  
Pressurized by:

Date:

Pressurization Gas:

N<sub>2</sub> He

Lab I.D.	Field Sample I.D. (Location)	Can #	Date of Collection	Time of Collection	Analyses Requested	Canister Pressure/Vacuum	Initial	Final	Receipt	Final (gall)
01A	B301A-1 101715	34753	10-18-15	1100	T0-15, CH4		28	6.5		
02A	B301A-2 101715	641270	10-18-15	1126	T0-15, CH4		29	6.5		
03A	B301A-3 101715	31486	10-18-15	1159	T0-15, CH4		30	8		
04A	B301A-4 101715	33898	10-18-15	1138	T0-15, CH4		27	5		
05A	B301A-4D 101715	12940	10-18-15	1138	T0-15, CH4		29	4		
06A	B301A-5 101715	916	10-18-15	1132	T0-15, CH4		29	6		
07A	B421A-1 101715	5681	10-18-15	1238	T0-15, CH4		29	6		
08A	B421A-2 101715	410017	10-18-15	0754	T0-15, CH4		30	5		
09A	B421A-3 101715	34746	10-18-15	0752	T0-15, CH4		30	3		
10A	B3042RA	901	10-18-15	1345	T0-15, CH4		29.5	8.5		
Relinquished by: (signature) <u>David Lindstrand</u> Date/Time <u>10-19-15 12:00</u>		Received by: (signature) <u>STALC</u> Date/Time <u>10-20-15 1035</u>		Notes: Report results to MDL Shipped via FedEx by AMA1 Tracking No. 77476748 2387						
Relinquished by: (signature) _____ Date/Time _____		Received by: (signature) _____ Date/Time _____								
Relinquished by: (signature) _____ Date/Time _____		Received by: (signature) _____ Date/Time _____								
Lab Use Only	Shipper Name <u>FedEx</u>	Air Bill # _____	Temp (°C) <u>18</u>	Condition <u>Good</u>	Custody Seals Intact? <u>Yes</u>	Work Order # <u>1510351</u>				



eurofins

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FOLSOM, CA 95630-4719  
(916) 985-1000 FAX (916) 985-1020

Page 2 of 2

Project Manager

Terry Taylor

Collected by: (Print and Sign)

David Lindstrand

Company

AMA1

Email

Address 2700 Westchester City Purchase State NY Zip 10577

Phone

914-251-0400

Fax

Project Info:

P.O. #

Project #

Project Name

BMS V1

Turn Around Time:

☐ Normal

☒ Rush

3-Day

Lab Use Only  
Pressurized by:

Date:

Pressurization Gas:

N<sub>2</sub> He

Lab I.D.

Field Sample I.D. (Location)

Can #

Date

Time

Analyses Requested

Canister Pressure/Vacuum

Initial

Final

Receipt

Final (gm)

11A

B01A-2 101715

GL1334

10-18-15

1145

TO-15, CH<sub>4</sub>, Napthalene

30

4.5

12A

B81A-2D 101715

342226

10-18-15

1145

TO-15, CH<sub>4</sub>, Napthalene

30

7

13A

B84A-1 101715

14881

10-18-15

1145

TO-15, CH<sub>4</sub>, Napthalene

30

4

14A

B301A-1 101715 F

5751

10-18-15

0806

Do Not Analyze

28

0

15A

10-19-15

10-19-15

Relinquished by: (signature) Date/Time

10-19-15 13:00

Received by: (signature) Date/Time

10/20/15 1:35

Notes:

Report results to MDL

Shipped via FedEx by AMA1

Tracking No.: 774767492387

Relinquished by: (signature) Date/Time

10-19-15 13:00

Received by: (signature) Date/Time

10/20/15 1:35

Relinquished by: (signature) Date/Time

10-19-15 13:00

Received by: (signature) Date/Time

10/20/15 1:35

Lab Use Only

Shipper Name

Air Bill #

Temp (°C)

Condition

Custody Seals Intact?

Work Order #

Lab Use Only

15/10351

## DATA REVIEW WORKSHEETS

Project Number: 1510351C

Date: 10/18/2015

### REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from ASTM D-1946 method for measuring permanent gases and light hydrocarbons in refinery and other sources samples using gas chromatography (GC) and a thermal conductivity detector (TCD) and/or flame ionization detection (FID). Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Eurofins data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: 1510351C

Sample matrix: Air

No. of Samples: 13

Trip blank No.: -

Field blank No.: -

Equipment blank No.: -

Field duplicate No.: B30IA-4\_101715/B30IA-4D\_101715; B8IA-2\_101715/B8IA-2D\_101715

☒ Data Completeness

☒ Holding Times

☐ N/A GC/MS Tuning

☐ N/A Internal Standard Performance

☒ Blanks

☐ N/A Surrogate Recoveries

☐ N/A Matrix Spike/Matrix Spike Duplicate

☒ Laboratory Control Spikes

☒ Field Duplicates

☒ Calibrations

☒ Compound Identifications

☒ Compound Quantitation

☒ Quantitation Limits

Overall Comments: Methane\_by ASTM\_method\_D-1946\_(modified)

#### Definition of Qualifiers:

J- Estimated results

U- Compound not detected

R- Rejected data

UJ- Estimated nondetect

Reviewer: Rafael Defaut

Date: 11/11/2015

## DATA REVIEW WORKSHEETS

## DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

### HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time				

### Criteria

Aqueous samples – 14 days from sample collection for preserved samples ( $\text{pH} \leq 2$ ,  $4^{\circ}\text{C}$ ), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples,  $4^{\circ}\text{C}$ , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria:  $4 \pm 2^{\circ}\text{C}$ ): N/A – summa canisters

### Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ).

If the % solid of soil samples is  $< 10\%$ , estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but  $< 14$  days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but  $< 28$  days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded ( $> 28$  days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ( $> 10^{\circ}\text{C}$ ), estimate positive results (J) and nondetects (UJ).

## DATA REVIEW WORKSHEETS

All criteria were met \_\_N/A\_\_  
Criteria were not met see below       

### GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

\_\_N/A\_\_ The BFB performance results were reviewed and found to be within the specified criteria.

\_\_N/A\_\_ BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List \_\_\_\_\_ the \_\_\_\_\_ samples \_\_\_\_\_ affected:

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If mass calibration is in error, all associated data are rejected.

Note: Samples analyzed using GC with either TCD or FID detection.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 05/15/15  
 Dates of continuing calibration: 10/22/15  
 Instrument ID numbers: GC-9  
 Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.					

#### Criteria

All RFs must be  $> 0.05$  regardless of method requirements for SPCC.  
 All %RSD must be  $\leq 15\%$  regardless of method requirements for CCC.  
 All %Ds must be  $\leq 30\%$  regardless of method requirements for CCC.  
 Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of  $\geq 0.995$  has therefore been utilized as professional judgment.

#### Actions

If any compound has an initial RF or a continuing RF of  $< 0.05$ , estimate positive results (J) and reject nondetects (R), regardless of method requirements.  
 If any compound has a %RSD  $> 15\%$ , estimate positive results (J) and use professional judgment to qualify nondetects.  
 If any compound has a %RSD  $> 90\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has a % D  $> 30\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has a % D  $> 30\%$ , estimate positive results (J) and nondetects (UJ).  
 If any compound has a % D  $> 90\%$ , estimate positive results (J) and reject nondetects (R).  
 If any compound has  $r < 0.995$ , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

#### V.A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

### Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

Field/Equipment/Trip blank

[illegible]



## DATA REVIEW WORKSHEETS

All criteria were met X  
Criteria were not met  
and/or see below \_\_\_\_\_

### V B. BLANK ANALYSIS RESULTS (Section 3)

## Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

**Specific actions are as follows:**

If the concentration is < sample quantitation limit (SQL) and  $\leq$  AL, report the compound as not detected (U) at the SQL.

If the concentration is  $\geq$  SQL but  $\leq$  AL, report the compound as not detected (U) at the reported concentration.

If the concentration is  $\geq$  SQL and  $>$  AL, report the concentration unqualified.

**Notes:**

### High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

## DATA REVIEW WORKSHEETS

All criteria were met \_\_N/A\_\_  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
-----------	--------------------	--------

\_\_\_\_Surrogate standards not required by the method.\_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

QC Limits\* (Air)

\_\_\_\_LL to UL\_\_\_\_to\_\_\_\_to\_\_\_\_to\_\_\_\_to\_\_\_\_

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

## DATA REVIEW WORKSHEETS

All criteria were met \_\_\_\_\_  
 Criteria were not met \_\_\_\_\_  
 and/or see below N/A \_\_\_\_\_

### VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

#### 1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ - \_\_\_\_\_ Matrix/Level: \_\_\_\_\_ - \_\_\_\_\_

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
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MS/MSD are not required as part of ASTM-method D-1946; blank spike used to assess accuracy

- \* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- \* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met \_\_\_\_\_  
Criteria were not met \_\_\_\_\_  
and/or see below N/A \_\_\_\_\_

### MS/MSD – Unspiked Compounds

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: \_\_\_\_\_ Matrix/Level/Unit: \_\_\_\_\_

[illegible]

**Actions:**

- \* If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).  
\* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

## DATA REVIEW WORKSHEETS

All criteria were met X  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

#### 1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?  
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
____LCS/LCSD_(Blank_spike)_analyzed_in_this_data_package;_recoveries_and_RPD_____			
____within_laboratory_control_limits._____			

\* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

\* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

#### 2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below           

### IX. FIELD/LABORATORY DUPLICATE PRECISION

Sample ID\_B30IA-4\_101715/B30IA-4D\_101715\_\_

Matrix:   Air  

Sample ID\_B8IA-2\_101715/B8IA-2D\_101715\_\_

Matrix:   Air  

Field/laboratory duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD  $\pm$  25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD for field duplicates within laboratory control limits. RPD for laboratory duplicate (LCS/LCSD) within laboratory control limits.					

#### Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

## DATA REVIEW WORKSHEETS

All criteria were met NA  
 Criteria were not met  
 and/or see below \_\_\_\_\_

### X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- \* Area of +40% or -40% of the IS area in the associated calibration standard.
- \* Retention time (RT) within  $\pm 0.06$  seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
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Internal standard not required by the method. Samples quantified by external standard method


Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

## DATA REVIEW WORKSHEETS

All criteria were met   X    
Criteria were not met  
and/or see below       

### XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

LCS

Methane                      RF = 157692659

$$[ ] = (1476338836)/(157692659)$$

$$= 9.362 \% \text{ OK}$$



## DATA REVIEW WORKSHEETS

All criteria were met   X    
 Criteria were not met  
 and/or see below       

### XII. QUANTITATION LIMITS

#### A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples diluted by a factor of less than 1.9		

#### B. Percent Solids

List samples which have  $\leq 50$  % solids

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#### Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)